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Indian Standard

PESTICIDES — DETERMINATION OF RESIDUES IN FOODS AND WATER — ENDOSULFAN

भारतीय मानक

कीटनाशी — खाद्य पदार्थों और पानी में अवशिष्ट ज्ञात करना — एंडोसल्फान

UDC 664 : 543 [632·951·028 END]

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

September 1989

Price Group 1

Pesticides Residue Analysis Sectional Committee, AFDC 56

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards on 23 February 1989, after the draft finalized by the Pesticides Residue Analysis Sectional Committee had been approved by the Agricultural and Food Products Division Council.

Endosulfan formulations are extensively used in agriculture for the control of several insect pests. This standard will enable the food, health authorities and others engaged in the field to follow a uniform test procedure for the estimation of endosulfan residues in various food commodities.

In the preparation of this standard, due consideration has been given to the limits of endosulfan residues laid under the provisions of *Prevention of Food Adulteration Rules, 1955* and the test method is sensitive to the prescribed level of residues.

In reporting the result of a test or analysis made in accordance with this standard, if the final value observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

PESTICIDES — DETERMINATION OF RESIDUES IN FOODS AND WATER — ENDOSULFAN

1 SCOPE

This standard prescribes the gas chromatographic method for the determination of endosulfan (1, 2, 3, 4, 7, 7 — hexachlorobicyclo—(2, 2, 1) — heptane— (2) bis — (oxymethylene) — 5, 6 — sulphite residues in foods (fruits, vegetables, oilseeds and animal products) and water.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
IS 1070 : 1977	Specification for water for general laboratory use (*second revision*)
IS 11380 : 1985	Method of sampling for the determination of pesticides residues in agricultural and food commodities

3 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4 SAMPLING

The representative samples for the purpose of estimating endosulfan residues in the samples shall be drawn in accordance with IS 11380 : 1985.

5 SAMPLE STORAGE

Samples shall be stored as such when the quantity is small or chances of its degradation do not exist. Otherwise, carry out extraction (*see* **8**) and store the extract. Depending upon the nature of the sample, keep the samples or their extracts either in deep-freezer at −15°C or in refrigerator until taken up for analysis. Ensure that the samples do not absorb or loose moisture during storage. Avoid undue long storage periods.

6 APPARATUS

6.1 Waring Blender or Equivalent

6.2 Kuderna Danish Evaporative Concentrator or Equivalent

6.3 Gas Chromatograph

Equipped with electron capture detector and operating under the following suggested parameters, which may be varied, according to the available facilities, provided standardization is done:

Column	Glass packed with 3 percent OV-17 on Chromosorb V (80-100 mesh)
Column oven temperature	190°C
Injection port temperature	220°C
Detector temperature	250°C
Carrier gas and flow rate	Nitrogen, 80 ml per minute

7 REAGENTS

7.1 ***n*-Hexane**, glass re-distilled.

7.2 Isopropyl alcohol, glass re-distilled.

7.3 Acetonitrile, glass re-distilled.

7.4 Darco G-60, or equivalent.

7.5 Sodium Sulphate, anhydrous.

7.6 Sodium Chloride, AR grade.

7.7 Acetone, glass re-distilled.

7.8 Standard Endosulfan Solution

Mixture containing *alpha* endosulfan, *beta* endosulfan and endosulfan sulphate, at the concentration of 1 μg/ml each, in *n*-hexane.

8 EXTRACTION

8.1 Fruits and Vegetables

Reduce 500 g of representative crop sample into small pieces using a food cutter. Weigh a representative portion of 50 g of the finely divided material into a high-speed waring blender. Add 150 ml of *n*-hexane-isopropyl alcohol mixture (2 : 1). Blend until the sample is thoroughly macerated. Filter the material by light suction on Buchner funnel. Re-extract the material again with the solvent. Filter again the material by light suction. Combine the solvent layers into a separatory funnel containing 100 ml of distilled water. Wash the isopropyl alcohol from the solution with three successive 100 ml volumes of water, discarding water each time. Drain the isopropyl alcohol free hexane into a flask containing sodium sulphate. Shake and allow to stand for 15 minutes. Filter off the sodium sulphate through filter paper and concentrate the hexane in Kuderna Danish evaporator.

8.2 Oilseeds, Nuts and Grains

Transfer the ground sample to a thimble and extract in soxhlet apparatus with *n*-hexane for 8 hours. Concentrate the *n*-hexane extract in Kuderna Danish evaporator and make the volume to 25 ml. Transfer *n*-hexane layer into a separatory funnel and shake it with 25 ml acetonitrile for 2 minutes and allow the phases to separate. Transfer the lower phase of acetonitrile to a second separatory funnel and re-extract the hexane phase with further (2 ml × 50 ml) acetonitrile. Combine the acetonitrile layers. Dilute the acetonitrile layer with 400 ml water and extract with (3 ml × 50 ml) *n*-hexane layers are combined and dried over anhydrous sodium sulphate.

8.3 Water

Filter 1 000 ml of water sample to remove particulate matter. Divide the filtrate into six separatory funnels and extract each portion in one order with the 100 ml *n*-hexane. Repeat the extraction procedure, extracting each portion in reverse order with a fresh 100 ml of *n*-hexane. Repeat this portion third time adding another 100 ml increment to all the portions progressively outward. Combine the three 100 ml hexane extracts in a flask. Dry over anhydrous sodium sulphate and concentrate in Kuderna Danish evaporator.

8.4 Animal Tissue

With a knife cut a representative 25 g tissue sample into small pieces, place it in a waring blender, add 75 ml of *n*-hexane-isopropyl alcohol mixture (2 : 1) and blend at low speed until the sample is thoroughly macerated. Filter the extract on the filter paper in a Buchner funnel. Wash the isopropyl alcohol with (3 ml × 50 ml) water. Dry the *n*-hexane extract over sodium sulphate and concentrate it in Kuderna Danish evaporator and make the volume to 25 ml. Extract the *n*-hexane layer with acetonitrile (3 ml × 25 ml). Combine the acetonitrile layers in a separatory funnel containiag 100 ml *n*-hexane and 500 ml saturated sodium chloride and exchange the acetonitrile portion into *n*-hexane as in **8.2.**

9 CLEAN-UP

Take 45 ml of *n*-hexane extract and add 5 ml of acetone. To this add 0·5 g of Darco G-60 or equivalent. Allow the flask to stand for 15 minutes with occasional shakings. Filter the extract, wash the residue with (3 ml × 15 ml) of *n*-hexane-acetone mixture (9 : 1), concentrate the extract and dissolve in *n*-hexane for analysis.

10 GAS CHROMATOGRAPHIC METHOD

10.1 Principle

The endosulfan residue extracted from the sample is estimated with the help of a gas chromatograph equipped with an electron capture detector. The endosulfan content is determined by comparing the response with the response for a known standard endosulfan of almost similar concentration.

10.2 Procedure

Inject 2 µl of solution obtained after clean up as in **9.1** into the column of the gas chromatograph. Identify the peaks for endosulfan *alpha*, endosulfan *beta* and endosulfan sulphate and measure the peak area.

10.3 Calculation

Endosulfan residue expressed in µg/g as the sum of endosulfan *alpha*, endosulfan *beta* and endosulfan sulphate.

$$\begin{array}{l}\text{Endosulfan } alpha/\\ \text{Endosulfan } beta/\\ \text{Endosulfan Sulphate,}\\ \mu g/g\end{array} = \frac{A_1 \times V_2 \times V_3 \times C}{A_2 \times V_1 \times M} \times f$$

where

A_1 = peak area of the sample;

A_2 = peak area of the standard;

V_1 = volume, in µl, of the sample injected;

V_2 = volume, in µl, of standard (endosulfan *alpha*/endosulfan *beta* and endosulfan sulphate) injected;

V_3 = total volume, in ml, of the sample solution;

C = concentration, in µg/g of the standard solution;

f = recovery factor = 100/percent mean recovery; and

M = mass, in g, of the sample taken for analysis.

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